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JULIE BILLINGSLEY
TEAM LEADER EXAMINATION
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PROVISIONAL SPECIFICATION

APPLICANT: **VARIAN AUSTRALIA PTY LTD**
Invention Title: **ELECTRODE OF MASS SPECTROMETRY**

The invention is described in the following statement:

ELECTRODE FOR MASS SPECTROMETRY

Technical Field

5 The present invention relates to an electrode for use in a reduced pressure region in a mass spectrometer whereby the electrode is subject to deposition of dielectric substances thereon. The electrode may be part of a mass analyser, ion optics system or ion guide, ion detector or source to spectrometer interface in a mass spectrometer, the mass spectrometer being used in conjunction with, for example, an inductively coupled plasma, 10 microwave induced plasma, liquid chromatograph, gas chromatograph or laser ablation.

Background

15 The following discussion of the background to the invention is included to explain the context of the invention. This is not to be taken as an admission that any of the material referred to was published, known or part of the common general knowledge in Australia as at the priority date established by the present application.

20 Electrodes within a reduced pressure region in a mass spectrometer which provide electric fields for forming or containing and propagating an ion beam, or for controlling the properties of an ion beam, or for mass filtration of ions, or for affecting other aspects of an ion beam relevant to the stable operation of a mass spectrometer, usually have smooth polished surfaces for 25 providing an equipotential boundary for an electric field. However such electrodes are subject to deposition of non-conducting (dielectric) substances thereon. Such dielectric deposits, which generally form a film, can arise from several sources including contaminants and chemically active species in ion beams representative of the composition of analytical samples presented to the mass spectrometer for analysis. Thus an ion beam that passes through a mass 30 spectrometer can include chemically active particles that can cause deposition of a dielectric film when they strike an electrode. The dielectric film can then cause build-up of electric charge on the surface of the electrode when charged particles contact the film. This surface charge causes unstable performance of

the mass spectrometer. Sometimes a chemically reactive residual gas present in the vacuum system of a mass spectrometer can initiate the film deposition process when the gas comes into contact with the surfaces of electrodes in the vacuum system. For example residual oil vapour (hydrocarbons) from vacuum pumps can initiate the growth of dielectric films on the surfaces of electrodes. The rate of accumulation of such films can be increased greatly when the deposition process is supplemented by ion and/or electron and/or photon bombardment of the affected surfaces. Such conditions are present in many mass spectrometers and are believed to be responsible for the deposition of dielectric films that very often can be found, for example, on the ion optics and on the fringe rods of a quadrupole mass analyser in an inductively coupled plasma mass spectrometer. Residual oil vapour accompanied by ion bombardment can produce hydrocarbon-based dielectric or semi-dielectric films on these components. These dielectric films can be highly detrimental to the stability of the instrument's performance.

An object of the present invention is to provide an electrode for use in a reduced pressure region in a mass spectrometer in which the likelihood of deposition of dielectric substances onto the electrode is reduced.

Disclosure of the Invention

According to the invention there is provided an electrode for use in a reduced pressure region in a mass spectrometer whereby the electrode is subject to deposition of dielectric substances thereon,

the electrode having a surface portion for providing an equipotential boundary of an electric field for influencing charged particles,

wherein the surface portion is rough thereby providing projections and cavities for reducing deposition of dielectric substances from the charged particles onto the surface portion.

The projections and cavities that provide the roughness of the surface portion of the electrode may have a periodical or regular occurrence and may be provided by, for example, cuts, threads, channels, holes or similar in the surface portion. Alternatively the projections and cavities may have a non-

periodical or irregular occurrence and may be provided by, for example, sandblasting, stoning or scratching treatments of the surface portion.

5 It has been found that deposition of a dielectric film is less likely to occur
when the surface portion of the electrode that defines an equipotential boundary
for an electric field is not polished as for prior art electrodes, but instead is made
rough by the inclusion or projections and cavities. A possible but as yet
unconfirmed explanation for this observation might be that when an electrode
10 surface exposed to a flux of potentially contaminating particles consists of a
combination of cavities and projections (which may be micro-cavities and micro-
pinnacles), then that surface is in a favourable condition for dispersing initial
deposits of contaminating film around the projections in such a way that at least
the projections tend to stay clean. As long as the projections are clean, the
electric field around the electrode remains stable and causes no change in the
15 performance of the mass spectrometer.

According to the invention, the "degree of roughness" of the surface may
be quite pronounced, for example a distance of approximately 0.5 mm from the
peak of a projection to the base of a cavity has provided significantly improved
20 results compared to a prior art "smooth" surface electrode.

Preferably the surface portion in question of an electrode according to
the invention is provided with a screw thread formation to provide the
roughness.
25

The invention extends to the provision of a mass spectrometer, or a
component thereof such as for example an ion guide or mass filter, which
includes an electrode according to the invention.

30 For a better understanding of the invention and to show how the same
may be put into effect, several embodiments thereof will now be described, by
way of non-limiting example only, with reference to the accompanying drawings.

Brief Description of Drawings

Figs. 1(A) and 1(B) are diagrammatic illustrations to assist a possible explanation of the observation upon which the invention is based (that is, how a rough electrode surface in a vacuum system of a mass spectrometer is less likely to have a dielectric film deposited on it compared to a smooth electrode surface).

Fig. 2(A) to (D) schematically illustrate cylindrical electrodes (that is, round rods), for example of a quadrupole mass filter, according to an embodiment of the invention.

Figs. 3(A) and (B) schematically illustrate a preferred embodiment of the invention, which is a threaded round rod electrode.

Figs. 4(A) and (B) schematically illustrate a periodical structure for a round rod electrode which may provide the rough surface.

Figs. 5 to 14 schematically illustrate rough surface portions of electrodes according to embodiments of the invention, wherein the roughness is provided by various periodical and non-periodical structures.

Detailed Description of Embodiments

It is known that dielectric film when deposited on electrodes in a vacuum system of a mass spectrometer can cause build-up of electrical charges on the affected surfaces. This causes changes in the electrical fields around the electrode causing changes in the performance characteristics of the mass spectrometer. The present invention is based on the observation that film deposition is less likely to happen when the surface is not polished, but is rough.

Figs. 1(A) and (B) illustrate a surface portion 22 of an electrode 20 for use in a reduced pressure region in a mass spectrometer. The surface portion 22 is rough thereby providing projections 24 and cavities 26. The projections 24 and cavities 26 of surface 22, it is thought, provide multiple conditions that help

to disperse a contaminating film build-up. These conditions include, surface electrostatic field gradient, surface molecular diffusion, localised electron emission (including secondary electron emission), angle of impact of the primary contaminant flux onto the projections 24 (wash-out effect), and ion impact density gradient onto the projections 24. All of these phenomena help to keep the projections 24 of the electrode surface 22 cleaner and therefore in working condition. Figs. 1(A) and (B) illustrate a flux 28 of potentially contaminating ions approaching the rough surface 22 of the electrode 20. The electric field produced in proximity to the rough surface 22 is not uniform, but rather is distorted having electric field density gradients 30. The projections 24 have a higher density electric field. This field may change the ion impact trajectory and/or energy near the projections 24. The projections 24 may produce excessive electron emission as the result of ion impact and excessive electric field, thus helping to desorb particles from the surface by Electron Stimulated Desorption. This would help to keep the surface 22 of the electrode 20 cleaner than the surface would be without having the projections 24 and cavities 26, that is, if the surface were polished. As shown in Fig. 1(B), when energetic ions 28 impact at 32 onto the angular surface 34 of the projections 24 and cavities 26, this produces a "flushing" effect along the surface 24 down to the cavities 26, helping to keep the projections 24 cleaner. This flushing effect could be enhanced by the molecular diffusion of the contaminants on the surface under the influence of the projections 24-cavities 26 surface field gradient resulting from the angled impact of the primary contaminant ion and the working electrode voltages.

25

Figs. 2(A), (B) and (C) illustrate a round electrode 32 having a rough surface portion 34. Figs. 2(A) and (B) show, respectively, a portion of a transverse and a longitudinal cross-section of the rod electrode 32. Fig. 2(D) shows a quadrupole ion guide 36 made up of four of the rods 32 wherein the rough surface portions 34 face the volume 38 between the electrodes 32 where ions 40 mainly exist and from which contaminants may come.

30

Fig. 3 shows a preferred embodiment of the invention, which involves a relatively simple way of providing a controlled rough surface on a rod electrode

42, namely by cutting a screw thread 44 around the rod electrode 42. Fig. 3(A) is a transverse cross-section of the rod 42. The inherent simplicity of this way of providing a rough surface and the well controlled mechanical tolerances that are possible with the cutting of screw threads makes this the preferred way of providing a periodically rough surface.

The resulting electrode structure of Fig. 3 has been applied to a set of quadrupole fringe electrodes as disclosed in International application No. PCT/AU01/01024 (WO 01/91159 A1). Each of the four electrodes in the set was 9 mm in diameter. Threads were cut over a 12 mm length at the end of each electrode that faced the incoming ions. The threads were of 0.5 mm pitch; the cross-section of each thread approximated an equilateral triangle, so the angle at the apex was 60 degrees. The apices of the threads were made as sharp as the machining process would permit. The electrodes were assembled as described in PCT/AU01/01024 for use in a quadrupole mass analyser in an inductively coupled plasma mass spectrometer. Previously, a similar set of electrodes without threads had been used in the same instrument. After the threaded electrodes were installed the instrument's analytical performance showed improved stability compared to that observed when the electrodes were not threaded. The unthreaded electrodes were associated with a gradual loss of analytical signal that could be restored temporarily by application of a negative DC potential to the electrode assembly in addition to the normal radio frequency voltage. Eventually the electrode assembly had to be removed and each electrode vigorously cleaned to remove deposited dielectric films. With the threaded rods there was no need to apply a negative DC potential to the set of electrodes and when such a potential was applied, it had no effect on the analytical signal. This indicates that the set of electrodes was having its intended effect of introducing the ions into the mass filtering section of the quadrupole mass analyser, without disturbances associated with the accumulation and charging of dielectric films. Furthermore, the threaded rods have never been cleaned despite the instrument having been operated for a period of time at least 15 times as long as that over which the unthreaded rods had been in use before they had to be cleaned.

Other possible structures for providing a rough surface portion on an electrode in accordance with the invention include the provision of circumferential channels such as channels 46 in a rod electrode 48 (see Figs. 4(A) and 4(B)). Such channels could be cut to provide different shapes, such as saw-toothed 50 and 52 (see Figs. 5 and 6) or scalloped 54 (see Fig. 7). Projections 56 having a flat top 58 (see Fig. 8), or randomly provided projections 60 and cavities 62 (see Fig. 9), or projections 64 with shaped cavities 66 therebetween (see Fig. 10), or specially shaped tops 68 of projections 69 (see Fig. 11) are expected to deliver anti-contamination performance similar to the previous embodiments. The figures demonstrate that surface irregularities of any shape create conditions favourable to preventing the accumulation of contaminating film. Fig. 9 illustrates that a rough surface can be inexpensively produced by means of sand blasting, stone rumbling or by any other mechanical process that provides a roughened surface. As shown in, respectively, Figs. 12 and 13, it is possible to achieve the anti-contaminating effect with channels 70 or 74 having different orientations on an electrode 68 or 72. Instead of radial channels 74 (as in Fig. 13) it is possible to have channels 70 of other shapes oriented along the length of the rod 68 (see Fig. 12) to reduce contamination in accordance with the invention. It is also possible to produce the desired anti-contamination effect by making a rough surface by means of laser or any other non-mechanical influence that can produce cavities or holes 76 on the electrode 78 surface (see Fig. 14) leaving "projections" therebetween.

Electrodes having a rough surface portion according to the invention, regardless of how that surface is produced, when in a mass spectrometer, will have a greater ability than prior art smooth electrodes to resist the accumulation of contaminating film and will therefore provide more stable electrical characteristics in the presence of potentially contaminating substances. Such electrodes in mass spectrometers (such as inductively coupled plasma mass spectrometers) provide more stable and reproducible electrical fields when operated under conditions that would otherwise favour contamination (bad vacuum, presence of hydrocarbons from pump oil, aggressive samples). This

provides better mass spectrometer detection limits, improved stability, less signal drift, and reduced maintenance.

5 An additional advantage of the invention is that the electrode surfaces of an ion guide or mass filter can be made sufficiently rough that photons or energetic particles can be reflected at an angle greater than the incidence angle and are thereby diffused away from an ion detector. Thus, making the surface of the electrodes rough instead of providing the conventional highly polished surface reduces the reflection of energetic neutral particles or photons into a
10 detector and provides greater diffuse scattering of energetic neutrals and photons away from the detector, thereby reducing the continuous background without loss of analytical sensitivity, and consequently improving analytical detection limits.

15 The invention is applicable not only to the fringe rods of a quadrupole mass analyser but to all sorts of multipole ion guides, all multipole mass analysers and to all known rod shapes including hyperbolic rods. It is also applicable to all known charged particle electrodes including ion optics, detectors and source-interface electrodes. Rough surfaces on the ion optical
20 elements, interface and detector parts prevent accumulation of contamination and therefore provide more stable and reproducible instrument performance and reduced maintenance.

The invention described herein is susceptible to variations, modifications
25 and/or additions other than those specifically described and it is to be understood that the invention includes all such variations, modifications and/or additions which fall within the spirit and scope of the above description.

30 DATED: 8 October, 2003
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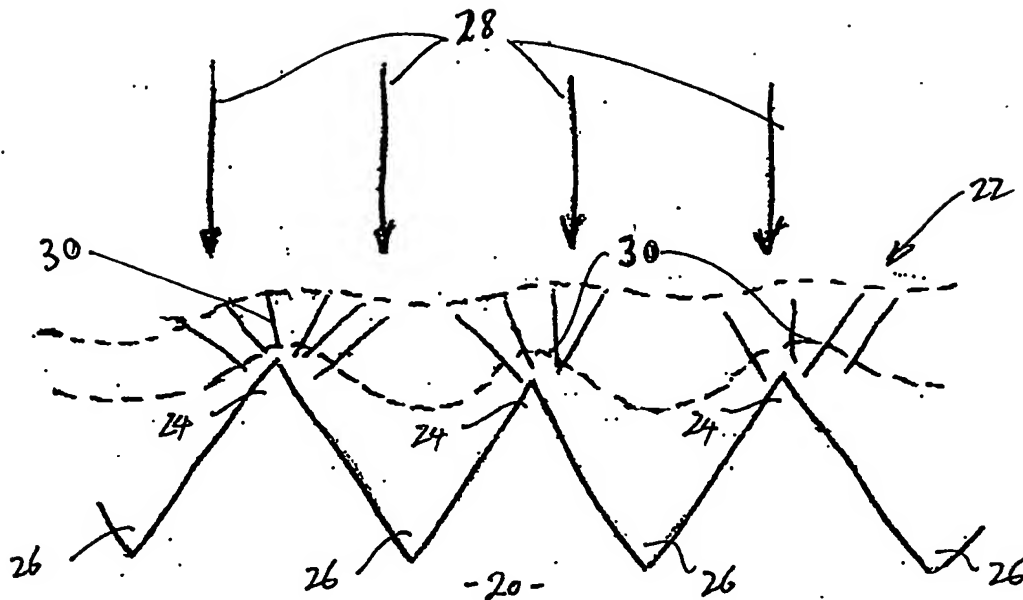


FIG 1A

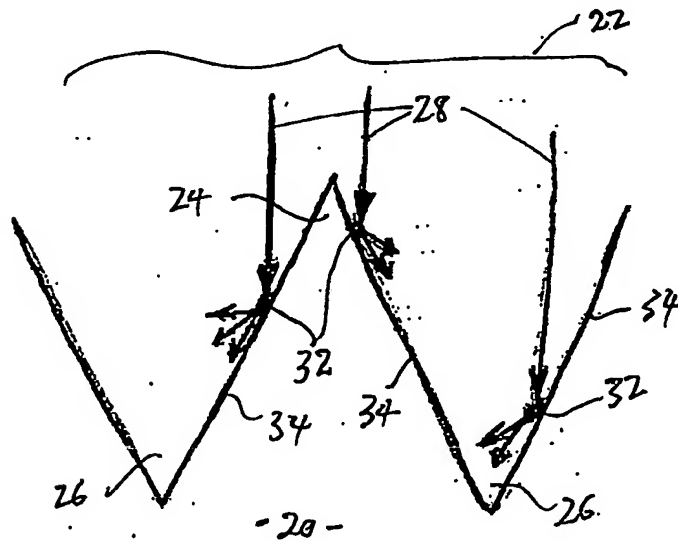
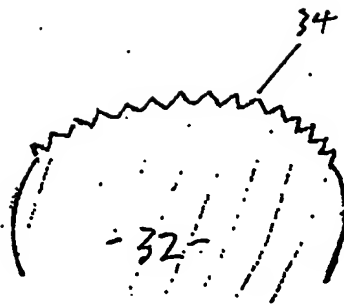
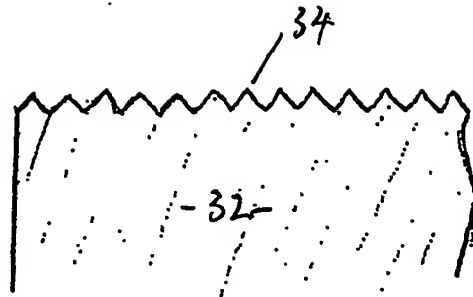


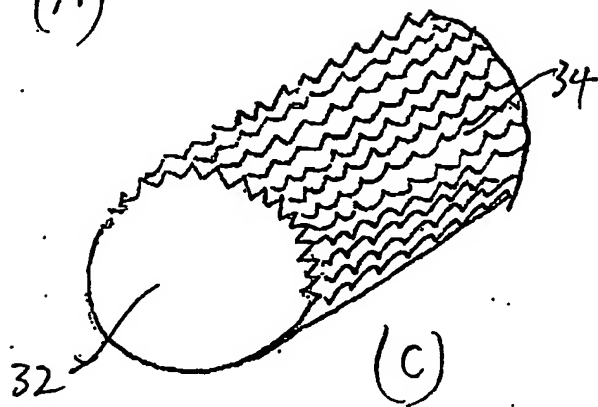
FIG 1B



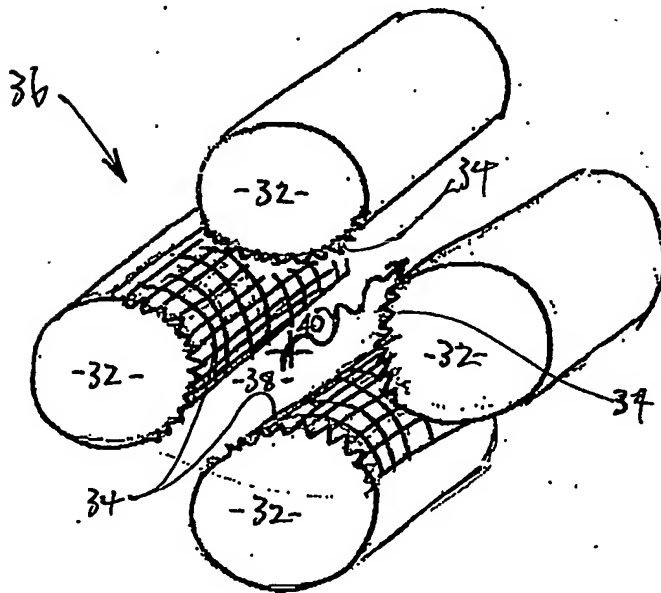
(A)



(B)



(C)



(D)

FIG 2

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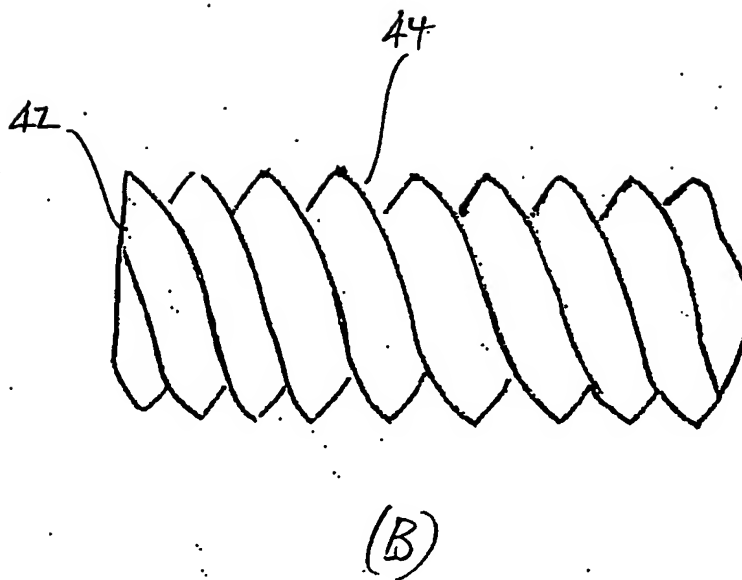
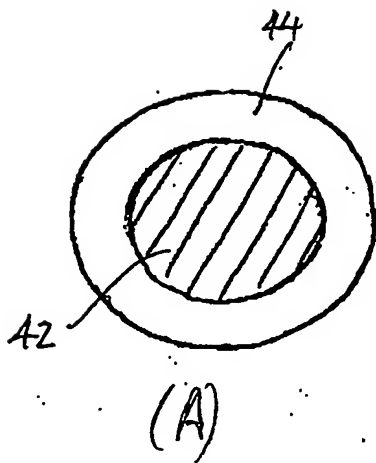


FIG 3

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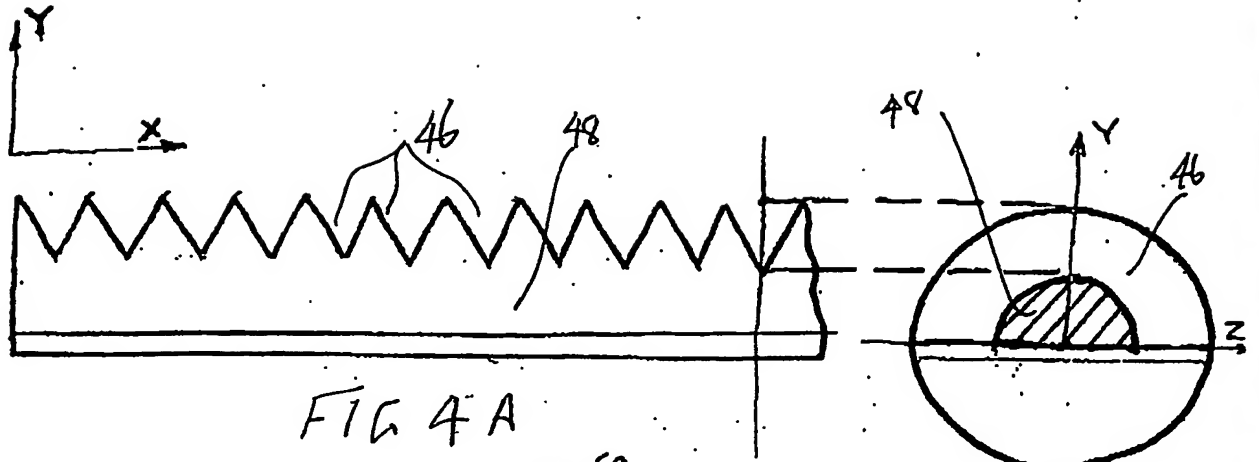


FIG 4A

FIG 4B

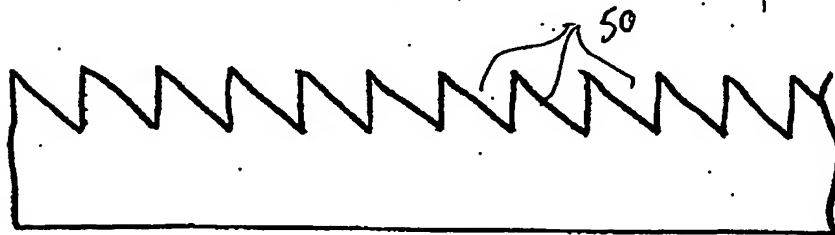


FIG 5

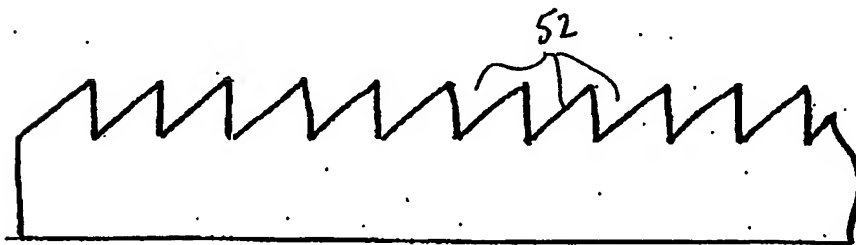


FIG 6

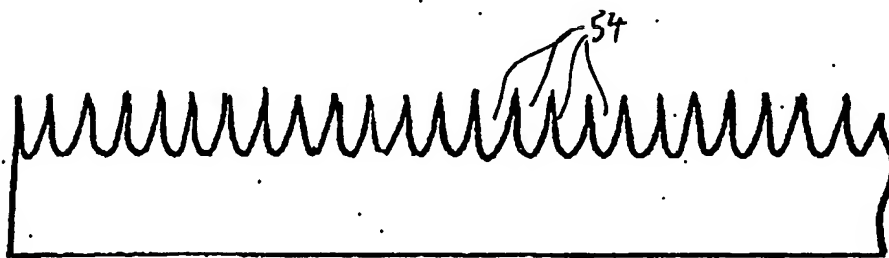


FIG 7

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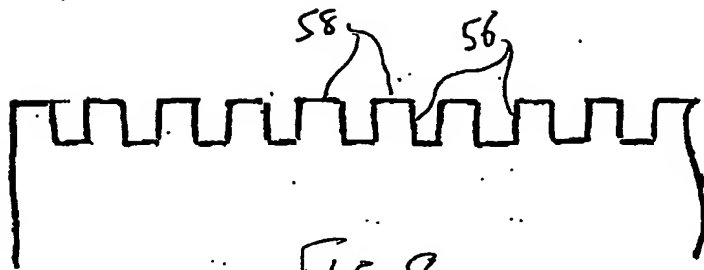


FIG 8

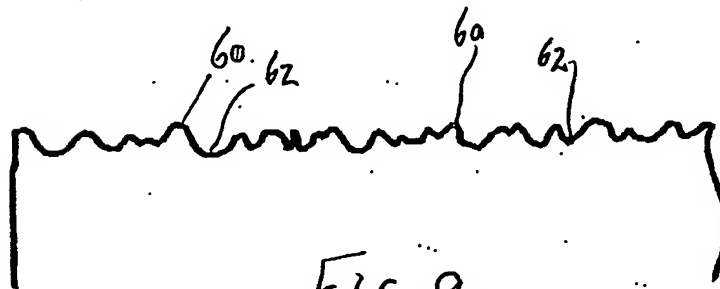


FIG 9

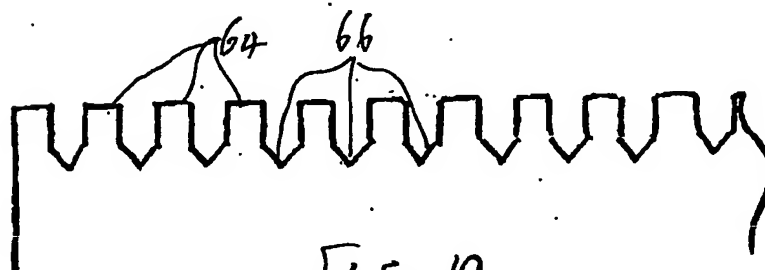


FIG 10

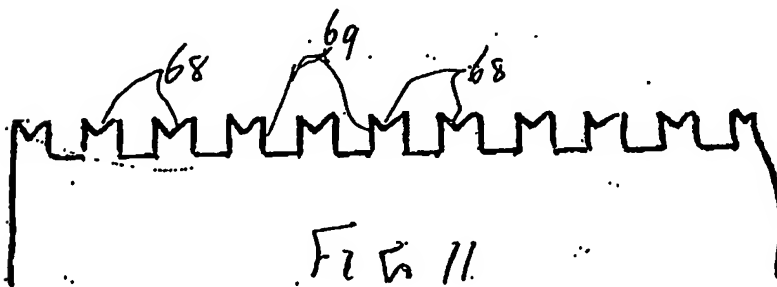


FIG 11

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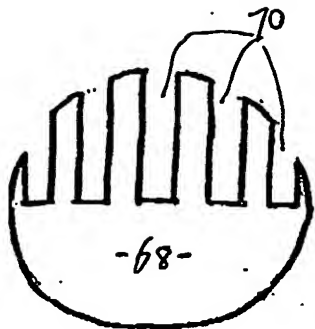
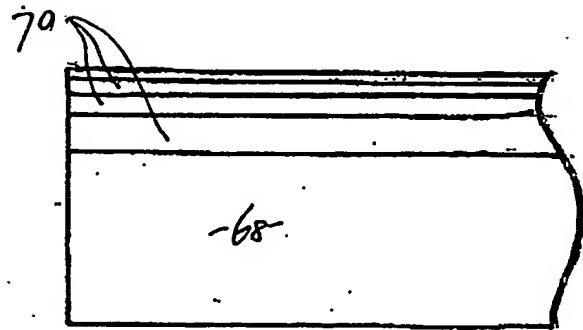


FIG 12 (A)



(B)

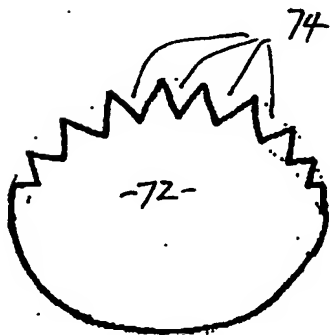
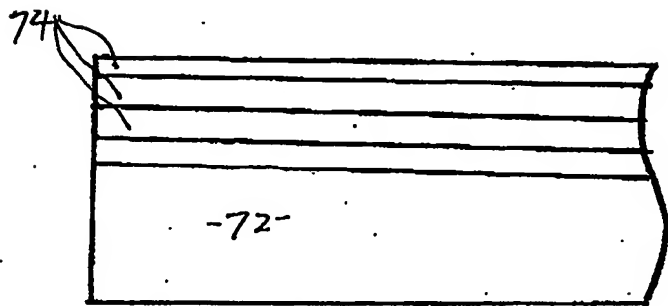


FIG 13 (A)



(B)

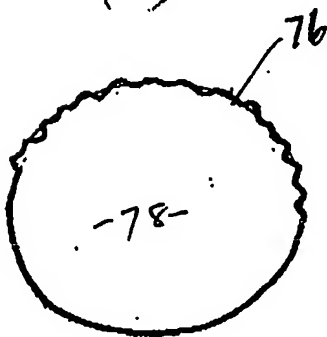
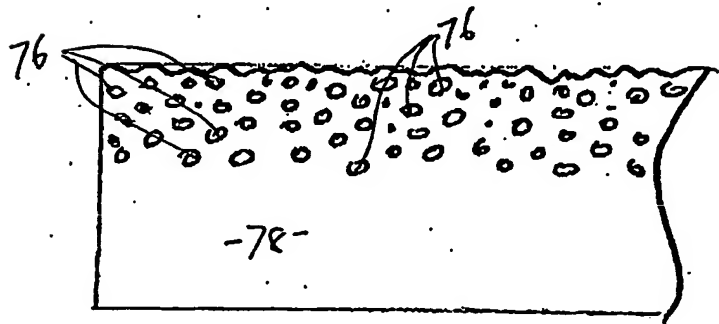


FIG 14 (A)



(B)

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